SUPERCRITICAL CARBON DIOXIDE EXTRACTION OF α-TOCOPHEROL FROM PALM LEAFLETS

Keywords :
Palm Leaflets; α-Tocopherol; Extraction; Supercritical Carbon Dioxide

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Extraction of α-tocopherol from palm leaflets by supercritical carbon dioxide (SC-CO_2) was attempted. The yields of SC-CO_2 extracts and α-tocopherol from dry samples were higher than those obtained from wet samples. It was also found that using dry samples at 200 kg/cm² and 40°C, it is possible to produce an extract containing more than 9% of α-tocopherol with a yield greater than 90%.

INTRODUCTION

Malaysia is the largest producer of palm oil in the world having about 1.9 million hectares of oil palm plantations in 1989. In order to optimize the economic performance of the industry, utilization of other products which are biosynthesized by the oil palm besides the oil needs to be investigated. Palm leaflets have been identified as a potential resource of α-tocopherol, carotenes and squalene (Ab Gapor *et al.*, 1986; Ab Gapor *et al.*, 1988). We are currently conducting research and development to establish suitable industrial technology for the extraction of valuable components from palm leaflets.

We considered that supercritical carbon dioxide (SC-CO_2) would be the most suitable solvent for α-tocopherol extraction: it is widely used for the extraction of thermally labile compounds and pharmaceuticals; it is non-toxic and non-flammable; and the method leaves no solvent residue in the product and operates at a low critical temperature, *i.e.* 31°C (Wong and Johnston, 1986; Larson and King, 1986). The present report is concerned with our efforts to apply the supercritical carbon dioxide technique to the extraction of α-tocopherol from palm leaflets.

MATERIALS AND METHODS

Palm Leaflets

Two samples of fresh, mature palm leaflets, A (moisture content, 57.4%) and B (moisture content, 60.9%) and four samples of chopped, freeze-dried palm leaflets C-F were obtained from the Palm Oil Research Institute of Malaysia (PORIM). The fresh palm leaflet samples (*ca.* 100g) were cut into small pieces using scissors and then ground in a coffee blender. The chopped, freeze-dried palm leaflets were treated in a similar manner.

Supercritical Carbon Dioxide Extraction Apparatus and Process

The apparatus used for the extraction has been described previously (Shishikura *et al.*, 1986; Zhao *et al.*, 1987) and a schematic diagram is shown in *Figure 1*. The samples were placed in the extractor (EX) which is a stainless steel cylinder (i.d., 4 cm, height, 20 cm) and which has a working pressure of 500 kg/cm² at 100°C. Pure CO_2 supplied from a cylinder (CY) was subjected to cooling (GC) and filtration (F), followed by compression (C) to the required pressure. After adjustment of the temperature by a heater (HE), the compressed CO_2 was passed through the extractor. The portion of the equipment enclosed by the dotted line in *Figure 1* was then immersed in a thermally regulated bath. The separation of CO_2 and the extract was achieved by depressurization of the SC-CO_2 via a metering valve (MV). The extract was collected in a receiver (RE). The volume of gas passing through the extractor was measured with a dry test meter (FM) and the flow rate was regulated at 6-10 NL/min. Addition of an entrainer to the SC-CO_2 before passing it through the extractor was also possible, by introducing a feeding pump to the apparatus.

Supercritical Carbon Dioxide Extraction of Palm Leaflets

A total of eight experiments were carried out, using different palm leaflet samples and conditions. For each experiment, about 20-50 g ground sample was placed in a cotton bag and introduced into the extractor. All the extractions were carried out at 40°C and at either 200 kg/cm² or 350 kg/cm² pressure. The use of either hexane or ethanol as an additive entrainer in the extraction was also investigated in three experimental runs. The weight of

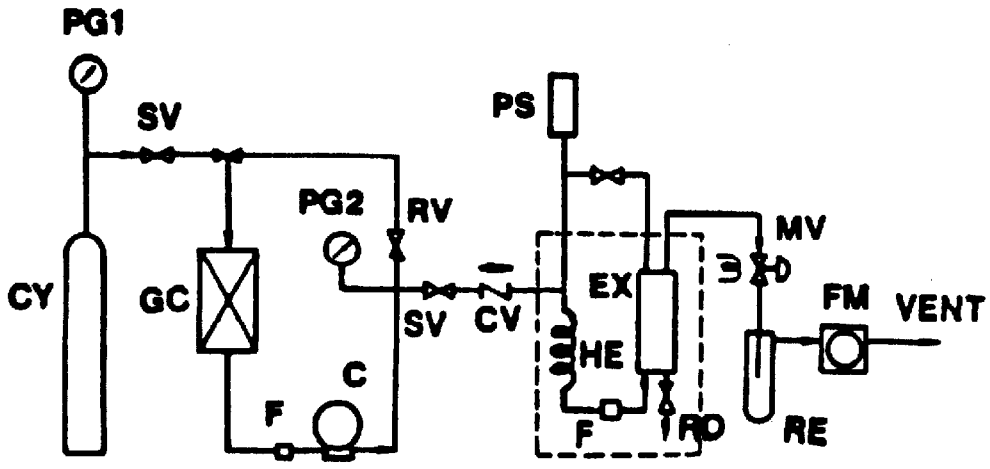

Figure 1. Schematic Diagram of SC-CO_2 Extraction Apparatus

CY, CO_2 cylinder; PGI, pressure gauge; GC, gas cooler; F, gas filter; C, compressor; RV, back pressure-regulating valve; PG2, precise pressure gauge; SV, shut-off valve; CV, check valve; PS, pressure sensor; HE, heater; EX, extractor; RD, rupture disk; MV, metering valve; FM, flow meter; RE, receiver.

the extract was recorded to facilitate determination of α-tocopherol content and yields.

Analysis of α-Tocopherol

The analysis of α-tocopherol in the feed sample for the supercritical CO_2 extraction and the corresponding extract was done by high performance liquid chromatography (HPLC). The HPLC system consisted of Micropump Type PHD-32 from Seishin Pharmaceutical Co., Injector KHP-ul-130 from Kyowa Seimitsu, Radial-PAK Cartridge (5 μm) silica column from Waters and, Model FP-210 Fluorescence Detector from Jasco (excitation 298 nm, emission 325nm). The eluting solvent used was 1% isopropyl alcohol in hexane and the flow was at 1.0 ml/min. 5-10 μl of sample solution was injected into the HPLC system and compared with α-tocopherol, which was used as an external standard. Preparation of a feed sample prior to injection into HPLC, which involved an extraction process using methanol-chloroform (2:1 v/v), was done in the manner described previously (Ab Gapor *et al.* 1986 ; Ab Gapor *et al.*, 1988). α-Tocopherol in the extract was analysed without additional treatment of the sample.

RESULTS AND DISCUSSION

The yield of the extract from supercritical carbon dioxide (SC-CO_2) extraction of palm leaflets is given in *Table 1*. It is obvious that the extraction yields from dry samples 2.0 - 2.6% were higher than those obtained from the wet samples, 0.41 and 0.71%. The extraction yields in the presence of hexane (two experiments) and ethanol as entrainers were 2.1%, 4.0% and 4.7% respectively, higher than those obtained without the entrainers.

The content and yield of α-tocopherol in SC-CO_2 extract are given in *Table 2*. The results showed that the yields of α-tocopherol in the extracts from wet samples (12.3 and 22.5%) were significantly lower than in those obtained from dry samples (65.8 - 93.8%). The α- tocopherol contents of the extracts

TABLE 1. YIELD FROM EXTRACTION OF PALM LEAFLETS WITH SUPERCRITICAL CARBON DIOXIDE

Expt. No.	Sample	Feed Amount (g)	SC-CO_2 Extraction Conditions			Yield	of Extract (%) *
			Temperature (°C)	Pressure (kg/cm²)	Entrainer	Consumption of CO_2 (NL)	
1	A(wet)	49.5	40	200	None	1764	1.71
2	B(wet)	30.7	40	350	None	2034	0.41
3	C(dry)	30.0	40	200	None	2157	2.0
4	D(dry)	19.9	40	200	Hexane (235 ml)	1663	2.1
5	E(dry)	30.1	40	200	Hexane (350 ml)	1749	4.0
6	E(dry)	30.3	40	200	Ethanol (300 ml)	2575	4.7
7	F(dry)	20.1	40	200	None	2181	2.3
8	F(dry)	19.5	40	350	None	1425	2.6

* Based on dry weight of feed

TABLE 2. CONTENT AND YIELD OF α -TOCOPHEROL IN SUPERCRITICAL CARBON DIOXIDE EXTRACT

Experiment No.	Sample	α-Tocopherol Content (%)*		α-Tocopherol Yield (%)
		Leaflet (Feed)	Extract	
1	A(wet)	0.19	2.6	22.5
2	B(wet)	0.23	6.74	12.3
3	C(dry)	0.09	2.88	65.8
4	D(dry)	0.13	5.04	78.2
5	E(dry)	0.44	10.33	93.8
6	E(dry)	0.44	8.03	84.8
7	F(dry)	0.23	9.27	92.4
8	F(dry)	0.23	8.10	92.0

* Dry basis

from wet samples were 2.6 and 6.74% and those obtained from dry samples ranged from 2.88 to 10.33%. Obviously, the α-tocopherol content in the extract was influenced by the initial level of α-tocopherol in the feed.

The effectiveness of using entrainers (hexane or ethanol) to increase α-tocopherol extraction efficiency was not clearly established in this limited number of experiments. There was an indication that an increase of CO_2 consumption resulted in the increases in the concentration and yield of α-tocopherol in the extracts. The effect of increasing extraction pressure from 200 kg/cm² to 350 kg/cm² was beneficial as while the consumption of supercritical CO_2 was low, the concentration and yield of α-tocopherol were relatively high.

CONCLUSION

It was found that α-tocopherol could be extracted from palm leaflets by using a supercritical CO_2 technique and that the extraction efficiency from dry samples was better than from wet. At 200 kg/cm² pressure, 40°C temperature, and with dry samples, the results showed that the α-tocopherol concentration in the extract could be >9% and its yield >90%. An increase in either supercritical CO_2 consumption or extraction pressure appeared to improve the overall performance of the extraction.

The use of entrainers such as hexane and ethanol was observed to be interesting from the viewpoint of isolating other components from palm leaflets (Ab Gapor, 1989). The SC-CO_2 extraction technique was thought to be attractive for the extraction of pharmaceutically useful α-tocopherol from palm leaflets. However, more studies need to be carried out in order to utilize the present results industrially.

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